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# DECONTAMINATION OF U-METAL SURFACE BY AN OXIDATION ETCHING SYSTEM

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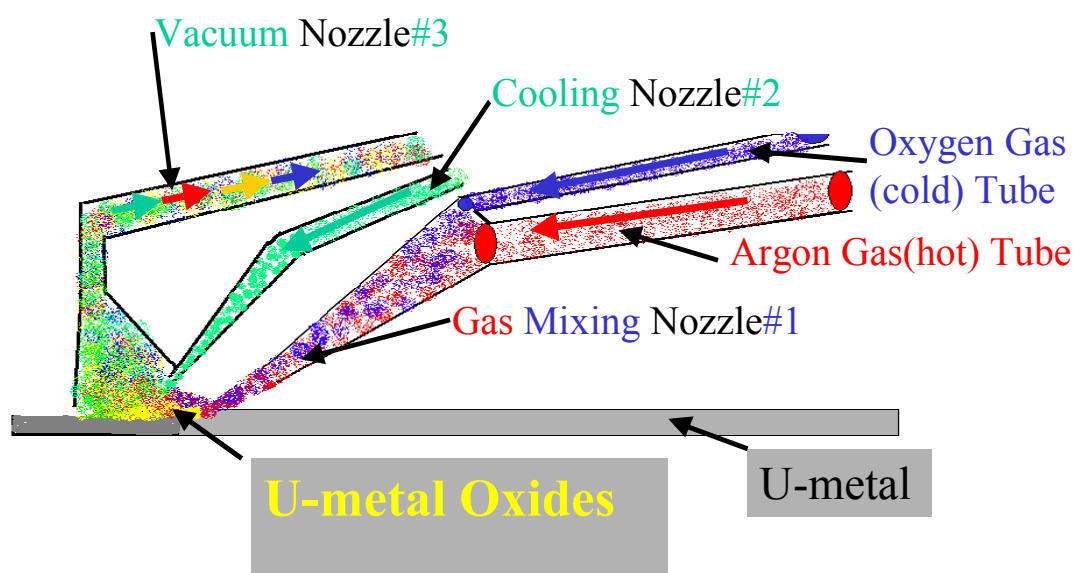
## INTRODUCTION

A surface oxidation treatment is described to remove surface contamination from uranium (U) metal and/or hydrides of uranium and heavy metals (HM) from U-metal parts. In the case of heavy metal atomic contamination on a surface, and potentially several atomic layers beneath, the surface oxidation treatment combines both chemical and chemically driven mechanical processes. The chemical process is a controlled temperature-time oxidization process that creates a thin film of uranium oxide ( $\text{UO}_2$  and higher oxides) on the U-metal surface. The chemically driven mechanical process is strain induced by the volume increase as the U-metal surface transforms to a  $\text{UO}_2$  surface film. These volume strains are sufficiently large to cause surface failure spalling/scale formation and thus, removal of a U-oxide film that contains the HM-contaminated surface. The case of a HM-hydride surface contamination layer can be treated similarly by using inert hot gas to decompose the U-hydrides and/or HM-hydrides that are contiguous with the surface. A preliminary analysis to design and to plan for a sequence of tests is developed. The tests will provide necessary and sufficient data to evaluate the effective implementation and operational characteristics of a safe and reliable system. The following description is limited to only a surface oxidation process for HM-decontamination.

The oxidation surface treatment process consists of a triple-nozzle system that moves over the surface of the U-metal at a controlled nozzle velocity relative to the U-metal. An illustration of a triple nozzle device is shown in Figure 1. The first nozzle will spray the U-metal surface with a mixture of oxygen (controlled fugacity) and inert (argon) gas (controlled gas flow rate) at a controlled oxidizing temperature (~200 to 450 C). Both U-metal and most other HM-metals of interest react rapidly with oxygen at these moderate temperatures and form higher metal-oxygen phases. These higher HM-oxides all have less density than the HM-metals [1,2]. Data for the U-metal oxidation rate indicate that at 300 C, the oxidation corrosion rate is ~ 1 micron/sec [3]. The second nozzle will spray the newly created oxide film with cold inert gas, at a controlled quenching temperature and gas flow rate, to quench the surface oxidation process.

Existing material property data [2,4] and models for time-dependent surface temperature due to a moving heat source [5,6] will be used to evaluate the spatial temperature distribution in the U-metal and any other HM-metals. The HM metal densities are  $\sim 17\text{g/cc}$ , and their first oxide phases are typically  $\text{HMO}_2$ , which nominally have significant decreases in densities to  $\sim 10\text{g/cc}$ . Thus, the incremental volume strain of this local phase change for the first HM-oxide phases is  $\sim 41\%$  [1,2,3] and for isotropic dilation strain components, each eigen-strain component would be  $\sim 14\%$ . This means that the formation of thin surface U-oxide films can not adhere to the U-metal substrate for high temperature-gradient induced phase transformations, and the film readily "scales or spalls" off and carries with it any HM-metal surface contamination. Finally, the third nozzle is a vacuuming pickup nozzle at a controlled gas flow rate for removing the spall and scaling surface particles of the U-oxide plus any other HM-oxides. Thus, the "chemical step" is to form the HM-oxide phases during a controlled heat-up and cool-down time interval. And the phase change density jump of the "chemical step" drives, intrinsically, the mechanical strain step of HM-surface removal by spallation. Initiation of analyses for more detailed model development and for experimental apparatus design to perform tests at values of operational variables prescribed in test matrices remains to be completed. An analysis of operational variables that follows provides an approximate model for depth control of the surface oxidation process and an associated set of test matrix plans [7] for the development and deployment of this surface oxidation approach for HM decontamination of U-metal surfaces.

**Figure 1. - Oxidation Uranium Surface Etching Illustration**



## Operational Model Development Description

The first nozzle (#1) is an “oxidation” nozzle that can move at a velocity relative to the U-metal surface and spray a moderate temperature (~200 to ~450 C) mixture of inert (argon) and oxygen gases onto the U-metal surface. In order to evaluate designs and the performance for nozzle (#1), a preliminary model is described for the removal of a prescribed depth  $d_o$  of U-metal as U-oxide. (Bold letters will be used to identify most variables because, as part of the approximate model development, they will become one dimensional data vectors in the definitions of test matrices of experiments to be performed). The depth  $d_o$  will be functionally controlled with seven operational variables of nozzle (#1) as well as the operational variables described later for the other two nozzles. For nozzle (#1), the control variables are the nozzle velocity  $v$ , nozzle height from the U-surface  $H_1$ , nozzle dimensions  $b_1$  in the velocity direction and  $a_1$  in the nozzle width direction, gas mixture temperature  $T_1$ , oxygen fugacity of the mixture  $P_1$ , and the gas mass flow rate  $M_1$ . In addition, information and data for U-metal from readily available literature [1,2,3,4] will be used to model material rate responses caused by the control variables of nozzle (#1). For example, a known analytical solution used in welding problems [5,6] will be used to estimate the spatial and time temperature distribution for heating and cooling sources moving over a U-metal plate. The model will also be used to prescribe test matrix conditions for a set of experiments to provide operational data on the performance of the oxidation nozzle. For this test matrix, the use of depleted U-metal samples is proposed, however, safety issues may impact proposed time schedules and surrogate metals can also be used in the operational testing. Preliminary calculations for quasi-steady moving temperature states indicate that a nozzle moving at a few centimeters per second is a working order-of-magnitude speed to attain temperature profiles that penetrate a temperature contour depth of a few millimeters. From the temperature contours and the relative nozzle speed, a time at temperature without cooling, for a generic point in the U-metal, can be estimated. However, at the present time the depth of the subsequent oxide phase change can only be roughly estimated from the temperature history at a spatial point during a heating temperature cycle. Hence, only a rough estimate of depth will be possible because the welding solution temperature does not address the complexities of either the chemical reactions during the oxidation of U-metal, that also provides an additional source of heat, nor the spatial phase change kinetics and deformational kinematics during surface spallation. Furthermore, these latter two complexities are not well understood and are not explicitly represented in the approximation model developed below.

The depth of oxide can be estimated by integrating, over an arbitrary time interval, the oxidizing velocity of an oxidation front propagating into the U-metal. The velocity of an oxidation front is an oxygen fugacity and temperature dependent Arrhenius rate process that can be typically expressed with the equation [2,3]:

$$v_o(P_1, T(x,t)) = V_o(P_1) \exp(-Q/RT(x,t)) \quad (1)$$

where  $V_o(P_1)$  is the oxygen fugacity function dependence,  $Q$  is the Arrhenius activation energy,  $R$  is the perfect gas constant, and  $T(x,t)$  is temperature. At any prescribed oxygen fugacity from nozzle (#1) and any temperature field  $T(x,t)$ , an

estimate for the oxide depth  $d_o$  as a function of fugacity and temperature can be made by the time integration of Eq. (1) for an arbitrary  $0 \sim \tau$  time interval as:

$$d_o(P_1, T, \tau) = \int_{0 \rightarrow \tau} V_o(P_1) \exp(-Q/RT(x,t)) dt \quad (2)$$

The implicit function  $d_o(P_1, T, \tau)$  is continuously dependent on each of the arguments  $(P_1, T, \tau)$ , therefore, a Taylor-like function expansion and the mean-value theorem of integral calculus can be used to approximate Eq. (2) as:

$$d_o(P_1, T, \tau) \approx \{ \Delta V_o(P_{1*}) / \Delta P_1 \exp(-Q/RT_*) \Delta P_1 + V_o(P_{1*}) (Q/RT_* T_*) \exp(-Q/RT_*) \Delta T + V_o(P_{1*}) \exp(-Q/RT_*) \} \tau \quad (3)$$

where the function increments  $\{\Delta P_1, \Delta T\}$  cover a function neighborhood of  $(P_1, T, \tau)$ , during the arbitrary time interval  $\Delta t$ , and  $\{P_{1*}, T_*\}$  are functions in their associated function intervals  $\{\Delta P_1, \Delta T\}$ .

As stated above, the time interval  $0 \sim \tau$  is arbitrary; but this is true only in a mathematical sense. First consider a test involving only the heating cycle and an observed/measured  $d_o$ , the operational variables of the first nozzle determine the time duration and spatial profiles of the temperature contours in the U-metal. Next, add a cooling nozzle during an experimental setting, then time interval  $\tau$  is a functional of the operational variables of the heating nozzle (#1) as well as those of the cooling nozzle (#2). In the following, potential influences on the observed depth  $d_o$  from dimensions  $b_1$  and  $b_2$  and from dimensions  $a_1$  and  $a_2$  for nozzles (#1) and (#2), as well as any influences from the operational variables of nozzle (#3), will be neglected. With these dimensional variables neglected, the cooling nozzle (#2) has three similar, and one slightly different, operational variables to those of nozzle (#1); namely, a height  $H_2$ , a gas temperature  $T_2$ , a gas mass flow rate  $M_2$ , and a separation dimension  $D_{12}$  between nozzles (#1) and (#2). Thus, the effective set for oxide depth control contains nine operational variables from those of nozzles (#1) and (#2), and is given by  $(v, P_o, H_1, T_1, M_1, H_2, T_2, M_2, D_{12})$ . Furthermore, the time interval  $\tau$  is assumed to be a continuous functional of these nine operational variables. Using this continuity assumption, the functional  $\tau$  can be written as a differential expansion in terms of increments of the operational variables from some initial fixed point, at which the value of  $\tau$  is identically zero. Thus, even though the mathematical details of this functional are not presently known nor well understood, there exists a linear approximation given by the following differential expression:

$$\begin{aligned} \tau(v, P_1, H_1, T_1, M_1, H_2, T_2, M_2, D_{12}) = & (\Delta\tau / \Delta v) \Delta v + (\Delta\tau / \Delta P_1) \Delta P_1 + (\Delta\tau / \Delta H_1) \Delta H_1 \\ & + (\Delta\tau / \Delta T_1) \Delta T_1 + (\Delta\tau / \Delta M_1) \Delta M_1 + (\Delta\tau / \Delta H_2) \Delta H_2 + (\Delta\tau / \Delta T_2) \Delta T_2 \\ & + (\Delta\tau / \Delta M_2) \Delta M_2 + (\Delta\tau / \Delta D_{12}) \Delta D_{12} \end{aligned} \quad (4)$$

Eq. (4) can be substituted into Eq. (3) to obtain a preliminary model for oxidation surface etching depth  $d_o$ , this is given by:

$$\begin{aligned}
d_o \approx & \{ \Delta V_o(P_1) / \Delta P_1 \exp(-Q/RT_1) \Delta P_1 + V_o(P_1) (Q/RT_1 T_1) \exp(-Q/RT_1) \Delta T_1 \\
& + V_o(P_1) \exp(-Q/RT_1) \} \{ (\Delta \tau / \Delta v) \Delta v + (\Delta \tau / \Delta P_1) \Delta P_1 + (\Delta \tau / \Delta H_1) \Delta H_1 \\
& + (\Delta \tau / \Delta T_1) \Delta T_1 + (\Delta \tau / \Delta M_1) \Delta M_1 + (\Delta \tau / \Delta H_2) \Delta H_2 + (\Delta \tau / \Delta T_2) \Delta T_2 \\
& + (\Delta \tau / \Delta M_2) \Delta M_2 + (\Delta \tau / \Delta D_{12}) \Delta D_{12} \}
\end{aligned} \tag{5}$$

where the function point ( $P_{1*}, T_{1*}$ ) and increment ( $\Delta T_{1*}$ ) are replaced with ( $P_1, T_1$ ) and ( $\Delta T_1$ ), respectively. The oxide depth expression of Eq. (5) is quadratic in the variables  $\Delta P_1$  and  $\Delta T_1$ , and has cross-product terms of these two variables with the remaining variables ( $\Delta v$ ,  $\Delta H_1$ ,  $\Delta M_1$ ,  $\Delta H_2$ ,  $\Delta T_2$ ,  $\Delta M_2$ ,  $\Delta D_{12}$ ). Given the fact that some, perhaps significant, uncertainty exists in the function of Eq. (1) for U-metal oxidation, from a strictly experimental viewpoint [7], there are twenty-seven coefficients plus a value for  $Q$  to evaluate in Eq. (5). However, by using available data on U-metal oxidation, and estimating values for the U-metal oxidation function and its derivatives, then the number of unknowns reduce to nine coefficients in the expansion of Eq. (4) for  $\tau$ . In this case, the simplest model would be to assume that the nine coefficients are a set of nine parameters of the etching depth model of Eq. (5), and their values can be determined by a statistical regression analysis [7,8] of experiment data.

In addition to those differential coefficients in Eq. (5) that can be estimated directly from available oxidation data [1,2,3] and the oxidation front velocity expression of Eq. (1), some of the other coefficients can be estimated from the available welding solutions [5,6]. These estimations would evaluate differentials of  $\tau$ , such as  $(\Delta \tau / \Delta H_1)$ ,  $(\Delta \tau / \Delta T_1)$ ,  $(\Delta \tau / \Delta H_2)$ ,  $(\Delta \tau / \Delta T_2)$ , and  $(\Delta \tau / \Delta D_{12})$ , by taking numerical variations in parameters of those solutions, such as ( $v$ ,  $q_{\text{heat}}$ ,  $q_{\text{cool}}$ ,  $D_{12}$ ), where the welding solution parameters  $q_{\text{heat}}$  and  $q_{\text{cool}}$  are the heating and cooling energy fluxes through nozzles (#1) and (#2), respectively. If a more detailed welding solution existed that had boundary condition parameters of height above the surface, gas/plasma temperatures, and mass flow rates of the heating and cooling gases, then the evaluation of their coefficients would appear to be almost explicit and directly applicable. However, the two complexities noted above due to the oxide phase formation and the spallation kinematics, if also included as parameters in the welding solution, could greatly influence the values obtained from a numerical variation of such parameters. In the latter case, the oxygen fugacity coefficient given by  $(\Delta \tau / \Delta P_1)$  would have measurable and significant influence on the time interval  $\tau$ . Since such elaborate solutions are not available, a following section will discuss a test matrix of experiments to provide independent sets of data such that empirically estimated values of these coefficients can be determined by statistical regression methods.

Finally, nozzle (#3) is the "vacuuming" nozzle that moves with and behind the "quenching" nozzle. The vacuuming nozzle collects the spalled oxidized U-metal layer into a air stream and transports it to a HEPA filter collection/storage unit. In the above development of Eq. (5), it was assumed that the operational variables of nozzle (#3) would not significantly influence the U-metal oxidation depth  $d_o$ . The operational variables for designing nozzle (#3) are control parameters of height above the surface,  $H_3$ , mixture plus inert gas plus spall particulate stream temperature,  $T_3$ , and gas mass flow rate plus spall particulate mass,  $M_3$ , and the spacing distance between the quenching and the vacuuming nozzle,  $D_{23}$ . Of these,

only the temperature  $T_3$ , which is coupled to the mass flow rate  $M_3$ , could reasonably have significant influence. For this reason, the design of nozzle (#3) should control temperature  $T_3$  such that its value is approximately the energy mixing temperatures of nozzles (#1) and (#2). This can be readily accomplished by controlling the gaseous mass flow  $M_3$  to be approximately the sum of  $M_1$  and  $M_2$ , then temperature  $T_3$  is approximately the energy mixing temperature of the heating and cooling nozzles. The  $M_3$  mass flow rate is controllable by a down stream pressure controller of the HEPA filter. The careful design and testing of nozzle (#3) is necessary to address concerns of HM-oxide particulate removal in order to maintain a “clean” and controlled environment in the HM-decontamination environment. Otherwise, there would be re-contamination of cleaned surfaces occurring during the process. The controlled oxidation/spallation surface removal process will have small HM-oxide particulate velocities in comparison to alternative surface removal techniques such as laser plasma-spall/shock-spall/ablation or erosion/ablation processes using high velocity gases or grits. In addition, the surface kinematics of oxide particulate velocities will be a functional of design control parameters ( $v$ ,  $P_1$ ,  $H_1$ ,  $T_1$ ,  $M_1$ ,  $H_2$ ,  $T_2$ ,  $M_2$ ,  $D_{12}$ ,  $H_3$ ,  $T_3$ ,  $M_3$ ,  $D_{23}$ ) described above in the nozzle designs. And of these parameters, the latter four, ( $H_3$ ,  $T_3$ ,  $M_3$ ,  $D_{23}$ ), are considered an independent subset of operational variables that can be used to evaluate and to optimize the “effectiveness” HM-oxide particulate removal and the “cleanliness” of the working decontamination environment. The working environment for deployment of a HM-decontamination system will most likely be a typical glove box enclosure.

### Test Matrix Development Description

The development for a test matrix of experiments will only be for the surface oxidation depth function of Eq. (5). By making some simplifying assumptions, the number of unknown coefficients was reduced to nine. In an ideal mathematical world, this would mean nine sets of measured data for the U-metal oxidation depth  $d_0$  at nine independent values of the operational variables ( $v$ ,  $P_1$ ,  $H_1$ ,  $T_1$ ,  $M_1$ ,  $H_2$ ,  $T_2$ ,  $M_2$ ,  $D_{12}$ ). However, in a realistic engineering approach, this means that a test envelope domain (interval of values for a variable) must be estimated for each variable, and some experiments performed at each prescribed point in the nine-space of the operational variables ( $v$ ,  $P_1$ ,  $H_1$ ,  $T_1$ ,  $M_1$ ,  $H_2$ ,  $T_2$ ,  $M_2$ ,  $D_{12}$ ). Performing a nine-space matrix of tests can be a large and expensive task unless the number of prescribed points of the test matrix is artfully selected. The three main criteria for selecting prescribed test points should consider [7,8];

- 1) relative accuracy of the measured data in different regions of the test envelope,
- 2) nature of the proposed model that will be functionally descriptive of the chemical, physical, and mechanical processes that will be observed and measured as data during the experiments, and
- 3) mathematical independence, for this case in a nine-space point sense, of a sufficient number of prescribed test points to do a valid statistical regression analysis of the measured data.

The two standard approaches to selecting prescribed values for test matrices are termed [7], *classical* and *factorial*. The classical approach is almost universally used by engineers, and is considered perfectly general for all applications [7]. The factorial approach is not always applicable, but if applicable, would have the greatest



accuracy for the smallest prescribed set of test points. Here, the classical approach of test matrix development is used because of the potential complexities from chemical reactions and deformational kinematics during surface spallation that may be observable during experiments. If any potential complexities should occur during testing, the classical approach of test matrix development to gather experimental sets of independent data assures that the test envelope is well covered. Also, the classical approach of test matrix development ensures that the data are gathered in a manner such that these data are statistically robust for regression analyses with an almost arbitrary choice of possible physical response models with unknown coefficients.

In this case of a nine-space, consider the nine-space as a nine coordinate Cartesian space, with a coordinate axis for each of the operational variables ( $v$ ,  $P_1$ ,  $H_1$ ,  $T_1$ ,  $M_1$ ,  $H_2$ ,  $T_2$ ,  $M_2$ ,  $D_{12}$ ). In this instance, a nine-space rectangular box that contains all values of the operational variables between a “highest” corner and a “lowest” corner has two to the nine power corners, which is 512 corners. This number of experiments is large just to evaluate nine unknown coefficients, as per the assumption for a simplified model, of Eq. (5). By considering nozzles (#1) and (#2) as always having the same height values, that is  $H_1 = H_2$ , and by considering nozzles (#1) and (#2) as always having the same mass flow rate values, that is  $M_1 = M_2$ , then the number of independent coordinates is reduced to seven. This reduces the above nine-space box to a seven space box with two to the seven power corners, which is 128 corners. This number of 128 experiments should be augmented with another subset of tests performed in a neighborhood of and at the center of the seven space box. The selection of this subset is based on an engineering judgement viewpoint of the model and also from a consideration for accuracy as given in criterion 1) above. In the model of Eq. (5), it is noted that the two temperatures,  $T_1$ ,  $T_2$ , and the oxygen fugacity,  $P_1$ , variables appear as quadratic terms. Thus, a three-space box for the three operational variables ( $T_1, T_2, P_1$ ) in a neighborhood of the center of the seven-space box would add two to the three power interior corners, or eight internal points, at which tests should be also performed under conditions that the other four operational variables of ( $v$ ,  $H_1=H_2$ ,  $M_1=M_2$ ,  $D_{12}$ ) be evaluated at the center point of the seven-space box. This develops a test matrix of 136 prescribed values of the operational variables of nozzles (#1) and (#2); subject to the constraints on height and mass flow rate dependence. These 136 prescribed test points will span the operational limits set by the test envelope, and are clearly independent. Also, these prescribed test points are selected almost independently of any functional relations between variables in an associated physical model. Therefore, the experimental data set obtained at these prescribed points should be robust enough to evaluate the nine coefficients of Eq. (5), as well as other statistical regression metrics for quality of model, significance of coefficients, and uncertainty and/or goodness of model with respect to these data.

## Systems Design and Summary

A descriptive discussion of the chemical and mechanical processes of oxidation surface etching provided sufficient detail to derive a preliminary model in terms of controllable operational variables of a three-nozzle device. The mechanistic chemical

and mechanical processes intrinsic to oxidation surface etching are sufficiently complex that a detailed model development effort is not a practical or expedient alternative at this time. In addition, by making several simplifying assumptions for terms in the analytical expression of the preliminary model, the number of unknown coefficients that will have to be evaluated from experimental data were greatly reduced. This reduction in the number of coefficients also decreased the number of tests that will provide the necessary and sufficient independent data sets for a valid statistical regression analysis.

A systems' combination of the three nozzles into a compact tool head that is "size" controlled to "oxidize surface etch" a given geometrical shaped part will be primarily a function of the width of the nozzle heads. Thus, the nozzle width dimension will be evaluated in conjunction with and is constrained by the given geometrical shaped parts to be surface clean. The geometry of the nozzles may be straight or curved, and nozzles should be easily interchangeable to match the surface profiles of the parts. The tool-head will be designed so that a hand held and operated etching operation is possible for those cases where the numbers of parts are small. In cases where the numbers of similar parts are large, than an automated machine tool design will be also possible for purposes of reduced workers exposure and increased safety of operations. Since the etching forces on the tool-head are small relative to standard machine tool chip or grinding operations, and depending on part geometry, a small drill head, lathe, and/or milling machine could be efficiently modified to hold and position the tool-head relative to the surface of the HM-contaminated part. An automated machine tool design would provide an increase in the rate of the part-per-time productivity and would reduce the health risks-per-part operational metrics.

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